Effect of Heat Treatment Temperature on Mechanical Properties and Micro Structure of DIN 1.2316 Steel

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Abstract
The effects of varying temperature of heat treatment of DIN 1.2316 steel to mechanical properties and microstructure have been investigated. The samples were heated by using electrical furnace at 820°C, 860°C, 900°C for 1 hour, after that the samples were quenched in water. Then heat treated and quenched samples were measured vickers hardness and impact strength, the microstructure of samples were observed using optical microscope. The test results show that with increasing heat treatment temperature can increase the value of vickers hardness. The hardness value before heat treatment was only 386.00 kgf /mm², but the hardness value of samples after heat treatment at 820°C, 860°C, 900°C are respectively 391.29 kgf /mm², 470.57 kgf /mm² and 488.52 kgf /mm². While the impact energy value tends to decrease with increasing heat treatment temperature, where the impact energy before heat treatment is 1550.00 J /mm², and impact energy value of samples after heat treatment at 820°C, 860°C, 900°C respectively are 1320.15 J /mm², 1110.35 J /mm² and 950.00 J /mm². The sample before heat treatment according to observation with optical microscope and XRD has ferrite (α-Fe) and pearlite (γ-Fe) phases. But the samples after the heat treatment process show ferrite and pearlite phase tends to decrease and emerging new phase martensite (α'-Fe). This martensite phase causes the material to become hard.

Keywords: DIN 1.2316 steel, heat treatment, quenching

1.0 Introduction
Process of heat treatment is a combination of timed heating and cooling applied to a particular metal or alloy in the solid state in such ways as to produce certain micro structure and desired mechanical properties (hardness, tensile strength and impact strength)[ Noor Mazni Ismail et al, 2016, Senthilkumar Y and Ajiboye T K, 2012]. There are some of heat treatment processes such as: annealing, normalizing, hardening and tempering. Annealing is the type of heat treatment most frequently applied in order to soften iron or steel materials and refines its grains due to ferrite-pearlite micro structure [Sanjib Kumar Jaypuria, 2009, Jon S. Magdeski et al, 2003]. Normalizing is heating process of steel at the austenitic temperature range and this is followed by air cooling. This treatment is usually carried out to obtain a mainly pearlite matrix, which results into strength and hardness higher than in as received condition. It is also used to remove undesirable free carbide present in the as-received sample [V. B.
da Trindade Filho et al, 2004]. Steels are normally hardened and tempered to improve their mechanical properties, particularly their strength and wear resistance. In hardening, the steel is heated to a temperature high enough to promote the formation of austenite, held at that temperature until the desired amount of carbon has been dissolved and then quench in oil or water at a suitable rate. Also, in the harden condition, the steel should have 100% martensite to attain maximum yield strength, but it is very brittle too and thus, as quenched steels are used for very few engineering applications (such as cutting tools, dies tools) [Palash Biswas et al, 2018, Daramola O O et al, 2010]. In the rapid developing industry, steels continue to gain wide use as prospective functional and structural materials because of their good soft-magnetic properties, high strength, good corrosion, and wear resistance coupled with relatively low material cost [Daramola O O et al, 2010]. There are two advantages of use of steel materials: it is abundant in the earth’s crust in form of Fe₂O₃ and little energy is required to convert it to Fe and it can be made to exhibit great variety of micro structures and thus a wide range of mechanical properties [Adnan Calik et al, 2010]. The reason for its importance is that it is a tough, ductile and cheap material with reasonable casting, working and machining properties, which is also amenable to simple heat treatments to produce a wide range of properties [Mst. Nazma Sultana et al, 2014]. DIN 1.2316 steel contains carbon about 0.38% weight, Chrome about 16.50% and 1.20% weight of Mo. The Application of DIN 1.2316 steel is generally for molding material for example plastic mold and dies mold. This type of steel was originally still soft, but after the process of formation into a mold product needs to be done process of increasing its hardness in order to be able to be used as a mold [Mst.Nazma Sultana et al, 2011, Puteri Zirwatul et al, 2017]. So this steel is said to be steel that can be hardened through heat treatment process. It is capable for heat treatment therefore will determine the extent of its application. The heat treatment process is very wide, especially for application as a mold, hence required hard material and the appropriate heat treatment process is hardening process. In the hardening process, the heating temperature determines the quality and hardness level, generally the heating process is carried out to austenite forming at about 800-1000°C and followed by rapid cooling process so that the austenite can turn into martensite [Rollett A. D. & Graef M. De, 2015]. Research on heat treatment for stainless steel metals has been widely practiced. The factors that affect the increase in mechanical properties are heating temperature, cooling speed and composition of the content in steel [Frank Czerwinski, 2012, Shashank Shekhar, 2014]. Tukur S.A et al,2014 stated in his research that heat treatment at temperature 1000°C and rapid cooling using water for stainless steel type steel with Cr content about 13% wt. , it can increase hardness from 35 HRC to about 40-50 HRC and increase ultimate tensile strength nearly 60%.

The objective of the present study is to investigate the effect of heat treatment (hardening) on the mechanical properties (impact energy and hardness) and microstructure of medium carbon steel. In addition, the aim was to find an optimum heat treatment and quenching conditions of DIN 1.2316 steel.
2.0 Material and methods

In this study DIN 1.2316 steel is selected as specimen for experiment of heat treatment, effects of variation temperature of heat treatment to mechanical properties and microstructure have been investigated. The chemical composition of as-received steel consists of C: 0.38%wt., Cr: 16.50% wt., Mo = 1.20%Wt and Fe: balance. All specimens firstly, were heat treated at 820°C, 860°C, 900°C and hold for 1 hour by using electrical furnace in atmospheric conditions and followed by water quenching. Heat treated and quenched samples were then tested for vickers hardness, impact strength and microstructure using micropscope optic and X-Ray Diffractometer (XRD).

2.1 Testing

The measurement of vickers Hardness was done by using Microhardness tester. The applied load for hardness testing was 200 g load and pressure time of 15 seconds. Impact test is done by Charpy method, the parameter of impact test is to measure the amount of potential energy absorbed (Impact Energy) by the sample until the fracture occurs. So the amount of energy absorbed will determine whether the material is brittle or ductile. The impact Charpy test was conducted according ASTM No E-23, the size of sample for impact Charpy Test is seen in Figure 1.

The value of impact energy and impact strength can be determined by equations:

Impact Energy (EI):

\[
EI = m \cdot g \cdot h \cdot (\cos \alpha - \cos \beta)
\]

Where \( EI \) = impact strength (joule), \( m \) = mass of pendulum (12.3 kg), \( g \) = gravity, \( h \) = length of pendulum, \( \alpha \) = start of angle and \( \beta \) = angle after impact

Impact Strength (IS):

\[
IS = \frac{EI}{A}
\]

Where \( IS \) = impact strength (joule/mm²) and \( A \) = are specimen under notch.

The analysis of microstructure was done by using optical microscope and crystal structure by using X-Ray Diffractometer XRD). The grinding surface of the samples were polished using Al\(_2\)O\(_3\) carried on a micro clothe before observation by using optical microscope. The crystalline structure of the specimens was made visible by etching using solution 2.5 % Nital solution on the polished surfaces. The etching process was done by dipping in 2.5 % Nital solution for 20 minutes at room temperature.

3.0 Results and discussion

The results of measurement of Vickers Hardness and Impact Energy (IE) are shown on Figure 1.
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The vickers hardness and impact energy curve as function of temperature heat treatment

![Graph showing hardness and impact energy vs temperature](image)

**Figure 1.** The vickers hardness and impact energy curve as function of temperature heat treatment

The hardness and IE value of sample before heat treatment are about respectively 386 Hv and 1550 J/mm². It can be seen that surface hardness of heat treated samples increases with growing heat treatment temperatures, the value of hardness increases from 391.29 Hv to 488.52 Hv. When compared to the results of Takur S.A et al, an increase in hardness is close to that, whereas Takur SA et al result in increased hardness of 40-50 HRC (390-510 Hv) after heat treatment at 1000°C for SS 304 steel material [Tukur S.A et al, 2014]. But IE value tends decreasing with increasing of heat treatment temperatures. In this case, the sample before heat treatment has low hardness value and high IE value, it means that sample before heat treatment is still ductile, but after heat treatment became brittle. When the materials have low hardness it will become ductile and it can be seen that the material has a high IE value. For a material that is brittle will tend to be harder and IE value tends to shrink [Shashank Shekhar, 2014]. The effects of the heat treatment process accompanied by the quenching process will cause the material to tend to be brittle and hard. As it can be seen in Figure 2, the morphology of the micro structure changed before and after heat treatment. The microstructure of untreated specimen or before heat treatment (Figure 2.a) showed a combination of ferrite (black) and pearlite (grey). The sample after heat treatment at 820°C had martensite microstructure with small amount of ferrite as shown in figure 2.b. With increasing heat treatment temperatures the martensite microstructure tends to increase as shown in the 2c and 2.d.

The ferrite (black) structure can still be seen in black in smaller size and only partially changed to martensite. During the heating process to austenite temperature above 800°C, the Ferite phase with BCC (body centered cubic) crystal structure is transformed into an austenite phase with FCC (face centered cubic) crystal structure, then at rapid cooling process occurs phase transformation from FCC to BCT (body centered tetragonal), where martensite phase has BCT structure [16]. With the martensite phase forming process there will be increased hardness of steel material [Ersoy Erişir and Oğuz Gürkan Bilir, 2013].
Figure 2. Microstructure of sample before and after heat treatment.

Figure 3 shows typical XRD patterns of samples before and after heat treatment by X-Ray Diffractometer, using Cu Kα radiation at room temperature.

Figure 3. XRD patterns of DIN 1.2316 steel samples heat treated at different temperature and water quenched.
The XRD result shows that there are three peaks at $2\theta=44.41^\circ$, $65.42^\circ$ and $81.87^\circ$ for sample before heat treatment, all peaks indicate $\alpha$-Fe as ferrite phase with FCC structure. Based on XRD analysis, Cr content of 10.75% does not cause any reaction between Fe and Cr or C with Cr for this steel. Because the role of Cr with content below 11% only as a ferrite stabilizer and these elements are more soluble in $\alpha$-phase [Shashank Shekhar, 2014. Tukur S.A et al, 2014]. But Chrome improves strength, hardness, wear resistance, harden ability and corrosion resistance [Shashank Shekhar, 2014]. Based on the Fe-Cr phase diagram and with a Chrom content of <11% then at the time of cooling formed ferriti phase, and the steel used is classified as ferritic stainless steel [Shashank Shekhar, 2014. Tukur S.A et al, 2014]. After the stages of heat treatment and water quenching process, there is a phase transformation. For heat treatment at $820 ^\circ$ C the peak xrd at $2 \theta$ = 81.87 will be shifted to $2 \theta$ = 82.05, this indicates a phase change from $\alpha$- Fe to $\alpha'$-Fe, where $\alpha'$-Fe is martensite phase with BCT structure. Sample after heat treatment 860$^\circ$C and water quenching has three peaks, the highest peak is $\alpha$-Fe as ferrite phase at $2\theta = 44.41^\circ$, and the other peaks are $\alpha'$-Fe as martensite at $2\theta = 65.6^\circ$ and $2\theta = 82.07^\circ$. The XRD patterns of sample after heat treatment 900 $^\circ$C and water quenching is similar with sample after heat treatment 860$^\circ$C. The effect of heat treatment and rapid cooling or water quenching could cause transformation in phase of $\alpha$-Fe to $\alpha'$-Fe. This can be known that there a shift diffraction angle (two $\theta$).

4.0 Conclusion

Based on experimental results that the hardening process DIN 1.2316 steel has been successfully done with variations of temperature heating and water quenching. The highest hardness value was achieved at 470,566 - 488,522 Hv at the heating temperature of 860$^\circ$C and 900$^\circ$C. Formation of martensite phase (alpha $''$-Fe) after heat treatment causing increased hardness in steel.

The effect of increasing hardness value of steel material will become brittle with marked decrease of impact energy value. Therefore DIN 1.2316 steel can be used for use that requires high abrasive resistance, such as gear, sprocket and others.

References


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